

**SAMPLING QA/QC WORK PLAN****CORNELL-DUBILIER ELECTRONICS SITE****CITY OF SOUTH PLAINFIELD, NEW JERSEY**

Prepared by

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Prepared for

U.S. Environmental Protection Agency

Region II - Removal Action Branch

Edison, New Jersey 08837


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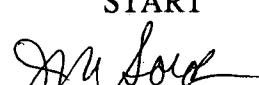
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Approved by:

START



Joseph J. Price
START Project Manager

Date: April 17, 1996

START


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Date: 4/17/96

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Date: 4/25/96

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1.

PROJECT DESCRIPTION

1.1. Site Background

The Cornell-Dubilier Electronics (CDE) site is located in South Plainfield, NJ in what is now known as Hamilton Industrial Park. CDE operated at this location from 1956 to 1961 testing transformer oils. It is alleged that during CDE's period of operation, the company dumped transformer oil contaminated with PCBs directly on site soils. Former employees have reportedly claimed that transformers were buried behind the facility during the same time period. Currently, approximately 15 businesses operate in a portion of the buildings formally occupied by CDE. The property is approximately 25 acres in size. The buildings occupy approximately 50% of the property. The remaining rear portion is comprised of an unused field and wetlands.

Sampling was conducted under the Pre-remedial program in June 1994 at the site. Of six soil samples collected from a depth of 0-1 ft, one sample revealed high concentrations of PCBs (1,100 ppm), lead (2,200 ppm), and cadmium (37 ppm). The area where this sample was collected is currently used by a truck driving school and is enclosed by a fence. During practice, drivers raise significant amounts of dust. PCBs were also detected in the other 5 samples collected in concentrations ranging from 68 ppm to 110 ppm.

1.2 Objective of Project

The objective of this project is to identify potentially hazardous substances, specifically lead, cadmium, and PCBs, that may become airborne through operation of heavy trucks operating within the fenced area of the site. The field program will include the collection of air samples at 4 locations. The sampling activities will be performed by the Roy F. Weston, Inc., Superfund Technical Assessment and Response Team (START) under the supervision of the U.S. EPA On-Scene Coordinator.

In order to confirm the presence of hazardous constituents in the air, the samples will be submitted to a subcontract laboratory for analysis. The analytical data will be used:

- to determine if off site migration of hazardous materials is occurring and at what concentrations
- to evaluate the potential threat to human health and the environment

- to determine whether the degree of threat meets EPA's criteria for a removal action
- if necessary, in the development of site removal/remediation plans

1.3 Scope of Work

The proposed sampling activities are scheduled for the week of April 22, 1996. A minimum of four air sampling locations will be identified to ensure that adequate information is gathered about the site. The 4 locations will include one upwind/background location; two site representative sample locations, and a sample location positioned downwind from the fenced area.

All air sampling locations will be identified on a map. The actual location of each air sampling point will be selected by the EPA OSC based upon likely contaminant exposure during typical dry site conditions. Two air samples will be collected at each sampling station. One will be collected for analysis for lead cadmium, silver, and arsenic using NIOSH Method 7300 (Elements, Revised 8/89), the second sample will be analyzed for PCBs using NIOSH Method 5503 (Poly-chlorobiphenyls). No duplicate samples will be collected at the direction of the EPA OSC.

Air sampling will be performed using the following criteria:

Parameter	Flow Rate	Sampling Period (minutes)	Calculated Sample Volume (Liters)
Lead, Cadmium Silver, Arsenic	3 L/minute	480	1440
PCBs	0.1 L/minute	480	48

Refer to Attachment B for standard operating procedures for the NIOSH methodologies used for this sampling event. All air samples will be submitted to a private subcontract laboratory for analysis.

1.4 Laboratory Analyses

All samples will be analyzed for the parameters specified in the "Scope of Work" section of this sampling plan. Additional information on the proposed analytical parameters is presented as Table 1. Sample analysis will be conducted in accordance with the quality assurance/quality control procedures presented in Sections 7 through 12 of this sampling plan.

TABLE 1 - ANALYTICAL PARAMETERS

Analytical Parameter	# of Samples	Sample Media	Analytical Method	Packaging	Holding Time	Calculated Sample Volume (L)
Lead, Cadmium Silver, Arsenic	4	MCEF* Filter	NIOSH 7300	ZIPLOCK BAG	180 DAYS	1440
Lot Blank	2		NIOSH 7300	ZIPLOCK BAG	180	0
Field Blank	2					
Matrix Spike Blank	2					
Polychlorobiphenyls (PCBs)	4	Florosil Sorbent tube + Glass fiber filter	NIOSH 5503	ZIPLOCK BAG	60 DAYS	48
Field Blanks	2		NIOSH 5503	ZIPLOCK BAG	180 DAYS	0
Media/Lot Blank	3					
Desorption Recovery	15					
Blind Spikes	3					
Analytical Spikes	3					

* MCEF = Mixed Cellulose Ester Filter

2.0 PROJECT ORGANIZATION AND RESPONSIBILITY

The following is a listing of key project personnel and their corresponding responsibilities:

Nicholas Magriples
U.S. EPA

On-Scene Coordinator

Joseph Price
Roy F. Weston, Inc.

START Project Manager
Sampler

Tamre Noblet
Roy F. Weston, Inc.

Sampler, Sample Packaging
Project QA/QC

3.0 **PROJECT FISCAL INFORMATION**

Air sampling filters, sorbent tubes, and sampling pumps will be provided by START. All man-hours expended by START will be charged to Technical Direction Document (TDD) # 96-04-0003. Laboratory analyses will be performed by a subcontracted laboratory acquired through the START contract.

4.0 **EQUIPMENT PREPARATION**

All air sampling equipment utilized in the course of this sampling event will be as specified for use in the specific sampling application by the NIOSH methodology. Separate sampling equipment will be dedicated to each sample location. Upon completion of sampling, air sampling pumps will be wiped clean using a slightly dampened cloth to minimize cross contamination of sampling areas.

Prior to air sampling, pumps will be individually calibrated using a rotameter previously calibrated against a bubble meter primary standard which was recently factory calibrated for precision and defensibility. Calibration records for each pump will be recorded in the field log book.

5.0 **SAMPLE LABELING**

Each sample will be accurately and completely identified. All labels will be moisture resistant and able to withstand field conditions. Sample containers will be labeled prior to sample collection. The information on each label will include, at a minimum, the following information:

- i. Site name or code
- ii. Sample number
- iii. Sample type (Elements or PCBs)
- iv. Date and Time of sample collection
- v. Person collecting the sample
- vi. Calculated sample volume in liters
- vii. Analysis to be performed by the laboratory

6.0 SAMPLE CUSTODY PROCEDURES

An EPA chain-of-custody record will be maintained throughout the sampling program as per EPA/ERT standard operating procedures (SOPs) on sample handling, and other applicable SOPs. The chain-of-custody SOP lists the following information at a minimum:

- i. Sample number
- ii. Number of sample containers
- iii. Description of sample, including specific location of sample collection
- iv. Identity of person collecting the sample
- v. Date and time of sample collection
- vi. Date and time of custody transfer to laboratory (if the sample was collected by a person other than laboratory personnel)
- vii. Identity of laboratory performing the analyses
- viii. Calculated sample volume (in Liters)

7.0 QUALITY ASSURANCE AND QUALITY CONTROL REQUIREMENTS

The level of quality assurance/quality control (QA/QC) as specified by the TDD, will be EPA QA Level 2 criteria.

In order to ensure accurate data, the following measures are required:

- a. Two unopened lot blanks will be supplied for the analysis for metals using NIOSH method 7300. The filters will be analyzed for lead, cadmium, silver and arsenic. For PCB analysis using NIOSH method 5503, 3 lot blanks of the florisil tubes and glass fiber filters will be analyzed as required by the method. The analysis of lot blanks will determine if contamination was imparted to the sampling media during the manufacturing process.
- b. One field blank is typically submitted per every 20 samples of each sampling media. For this sampling event, there will be two MCE filter field blanks, and two Florisil sorbent tube/glass fiber filter field blanks submitted as required by the respective NIOSH methods. A field blank sample is an opened tube or filter which travels throughout deployment of the sample pumps.

The field blank is designed to detect sample contamination that potentially occurs during field operations or during shipment. The field blank must be associated with an actual sampling event. ¹

- c. As required by the NIOSH 7300 (Elements), 2 matrix spike blanks will also be submitted for analysis for cadmium, lead, silver, and arsenic.

Per NIOSH method 5503 (PCBs), 15 desorption recovery blanks will be submitted, 3 blind spike samples, and 3 analytical spike blanks.

- d) The contracted laboratory will furnish the following deliverables as warranted:
- 1) GC/MS tuning and calibration standards
 - 2) Copies of all spectral data obtained during performance of analysis. Copies should be signed by the analyst and checked by the laboratory manager
 - 3) Data system printout (quantitation report or legible facsimile (GC/MS)
 - 4) Manual work sheets
 - 5) Identification and explanation of any analytical modifications that differ from the specified NIOSH methodology

All analytical results will be submitted by the laboratory to START. A written report will be submitted within 7 calendar days of the validated time of sample receipt (VTSR).

8.0 DOCUMENTATION, DATA REDUCTION AND REPORTING

Field data will be entered into a bound notebook. Field notebooks, chain-of-custody forms, and laboratory analyses reports will be filed and stored as per the START Document Control System.

9.0 DATA VALIDATION

All steps of data generation and handling will be evaluated by the START QA Officer for compliance with the specified requirements.

10.0 **SYSTEM AUDIT**

The Project QA/QC Officer will ensure the sampling operations are conducted in accordance with this sampling QA/QC work plan.

11.0 **CORRECTIVE ACTIONS**

All provisions will be taken in the field and laboratory to ensure that any problems that may develop will be dealt with as quickly as possible to ensure the continuity of the sampling program. Any deviations from this sampling plan will be noted in the final report.

12.0 **REPORTS**

A trip report describing the sampling activities will be submitted to EPA within five business days of the sampling event. Preliminary laboratory results and all requested QA/QC information will be submitted to EPA upon receipt of sample analyses. The final sampling report will be issued after receipt and validation of laboratory results.

BIBLIOGRAPHY

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ATTACHMENT A

SITE FIGURES



WESTON
MAINTAINING THE SCENE FOR CONSERVATION

Roy F. Weston, Inc.
FEDERAL PROGRAMS DIVISION

IN ASSOCIATION WITH RESOURCE APPLICATION INC.
C.C. JOHNSON & MALHOTRA, P.C. R.E. SARRIERA ASSOCIATES
PRC ENVIRONMENTAL MANAGEMENT, AND GRB ENVIRONMENTAL SERVICES, INC.

EPA PM

N. Magriples

START PM

Site Location Map

J. Price

Cornell-Dubillier Site

ATTACHMENT B

AIR SAMPLING METHODOLOGIES

NIOSH 7300 (ELEMENTS)

NIOSH 5503 (POLYCHLOROBIPHENYLS)

ELEMENTS by ICP

7300

MW: Table 1

CAS: Table 2

RTECS: Table 2

METHOD: 7300, Issue 2

EVALUATION: PARTIAL

Issue 1: 15 May 1989

Issue 2: 15 August 1994

OSHA: Table 2

NIOSH: Table 2

ACGIH: Table 2

PROPERTIES: Table 1

ELEMENTS: aluminum* chromium* lithium* phosphorus tellurium zinc
 arsenic cobalt* magnesium platinum* thallium zirconium*
 beryllium* copper manganese* selenium titanium
 cadmium iron molybdenum* silver vanadium
 calcium lead* nickel sodium yttrium

* Some compounds of those elements require special sample treatment.

SAMPLING		MEASUREMENT	
SAMPLER:	FILTER (0.8- μ m, cellulose ester membrane)	TECHNIQUE:	INDUCTIVELY COUPLED ARGON PLASMA, ATOMIC EMISSION SPECTROSCOPY
FLOW RATE:	1 to 4 L/min	ANALYTE:	elements above
VOL-MIN: -MAX:	Table 1 Table 1	ASHING REAGENTS:	conc. HNO ₃ , 4 mL; and conc. HClO ₄ , 1 mL
SHIPMENT:	routine	CONDITIONS:	room temperature, 30 min; 150 °C to near dryness
SAMPLE STABILITY:	stable	FINAL SOLUTION:	4% HNO ₃ , 1% HClO ₄ , 10 mL
BLANKS:	2 to 10 field blanks per set	WAVELENGTH:	depends upon element; Table 3
ACCURACY		BACKGROUND CORRECTION:	spectral wavelength shift
RANGE STUDIED:	not studied	CALIBRATION:	elements in 4% HNO ₃ , 1% HClO ₄
BIAS:	none identified	RANGE:	2.5 to 1000 μ g per sample [1]
OVERALL PRECISION (S_{γ}):	not evaluated	ESTIMATED LOD: 1 μ g per sample [1]	
ACCURACY:	not determined	PRECISION (S_{γ}):	Table 3

APPLICABILITY: The working range of this method is 0.005 to 2.0 mg/m³ for each element in a 500-L air sample. This is simultaneous elemental analysis, not compound specific. An alternative microwave digestion procedure is included. Verify that the types of compounds in the samples are soluble with the ashing procedure selected.

INTERFERENCES: Spectral interferences are the primary interferences encountered in ICP-AES analysis. These are minimized by judicious wavelength selection, interelement correction factors and background correction [1,2].

OTHER METHODS: This method replaces P&CAM 351 [2] for trace elements. Flame atomic absorption spectroscopy (e.g., Methods 7000) is an alternate analytical technique for many of these elements. Graphite furnace AAS (e.g., 7102 for Be, 7105 for Pb) is more sensitive.

REAGENTS:

1. Nitric acid, conc., ultra pure.
2. Perchloric acid, conc., ultra pure.*
3. Ashing acid: 4:1 (v/v) HNO_3 : HClO_4 . Mix 4 volumes conc. HNO_3 with 1 volume conc. HClO_4 .
4. Calibration stock solutions, 1000 $\mu\text{g/mL}$. Commercially available, or prepared per instrument manufacturer's recommendation (see step 12).
5. Dilution acid, 4% HNO_3 , 1% HClO_4 . Add 50 mL ashing acid to 600 mL water; dilute to 1 L.
6. Argon.
7. Distilled, deionized water.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: cellulose ester membrane filter, 0.8- μm pore size, 37-mm diameter; in cassette filter holder.
2. Personal sampling pump, 1 to 4 L/min, with flexible connecting tubing.
3. Inductively coupled plasma-atomic emission spectrometer, equipped as specified by the manufacturer for analysis of elements of interest.
4. Regulator, two-stage, for argon.
5. Beakers, Phillips, 125-mL, or Griffin, 50-mL, with watchglass covers.**
6. Volumetric flasks, 10- and 100- mL**
7. Assorted volumetric pipets as needed.**
8. Hotplate, surface temperature 150 °C.

** Clean all glassware with conc. nitric acid and rinse thoroughly in distilled water before use.

SPECIAL PRECAUTIONS: Perform all perchloric acid digestions in a perchloric acid hood.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Sample at an accurately known flow rate between 1 and 4 L/min for a total sample size of 200 to 2000 L (see Table 1) for TWA measurements. Do not exceed a filter loading of approximately 2 mg total dust.

SAMPLE PREPARATION:

3. Open the cassette filter holders and transfer the samples and blanks to clean beakers.
4. Add 5 mL ashing acid. Cover with a watchglass. Let stand 30 min at room temperature.
NOTE: Start a reagent blank at this step.
5. Heat on hotplate (120 °C) until ca. 0.5 mL remains.
NOTE 1: Recovery of lead from some paint matrices may require other digestion techniques. See Method 7082 (Lead by Flame AAS) for an alternative hotplate digestion procedure or the Appendix for a microwave digestion procedure [8].
NOTE 2: Some species of Al, Be, Co, Cr, Li, Mn, Mo, V, and Zr will not be completely solubilized by this procedure. Alternative solubilization techniques for most of these elements can be found elsewhere [2-7]. For example, aqua regia may be needed for Mn [4,9].
6. Add 2 mL ashing acid and repeat step 5. Repeat this step until the solution is clear.
7. Remove watchglass and rinse into the beaker with distilled water.
8. Increase the temperature to 150 °C and take the sample to near dryness (ca. 0.5 mL).
9. Dissolve the residue in 2 to 3 mL dilution acid.
10. Transfer the solutions quantitatively to 10-mL volumetric flasks.
11. Dilute to volume with dilution acid.

CALIBRATION AND QUALITY CONTROL:

12. Calibrate the spectrometer according to the manufacturers recommendations.

NOTE: Typically, an acid blank and 10 µg/mL multielement working standards are used. The following multielement combinations are chemically compatible in 4% HNO₃/1% HClO₄:

- a. Ag, Ca, Co, Mn, Pb, V, Zn;
- b. Al, Be, Cd, La, Li, Ni, Ti;
- c. As, B, Ba, Mg, Mo, P;
- d. Cu, Fe, Na, Pt, Sr, Te, Y;
- e. Cr, K, Se, Ti, Zr; and
- f. Si, W (distilled water only)

13. Analyze a standard for every ten samples.
14. Check recoveries with at least two spiked media blanks per ten samples.

MEASUREMENT:

15. Set spectrometer to conditions specified by manufacturer.
16. Analyze standards and samples.

NOTE: If the values for the samples are above the range of the standards, dilute the solutions with dilution acid, reanalyze and apply the appropriate dilution factor in the calculations.

CALCULATIONS:

17. Obtain the solution concentrations for the sample, C_s (µg/mL), and the average media blank, C_b (µg/mL), from the instrument.
18. Using the solution volumes of sample, V_s (mL), and media blank, V_b (mL), calculate the concentration, C (mg/m³), of each element in the air volume sampled, V (L):

$$C = \frac{C_s V_s - C_b V_b}{V}, \text{ mg/m}^3.$$

EVALUATION OF METHOD:

Method P&CAM 351 was evaluated in 1981 [1,2]. The precision and recovery data were determined at 2.5 and 1000 µg of each element per sample on spiked filters. The precision and recovery data, instrumental detection limits, sensitivity, and analytical wavelengths are listed in Table 3. The values in Table 3 were determined with a Jarrell-Ash Model 1160 ICP operated according to manufacturer's instructions.

REFERENCES:

- [1] Hull, R.D. "Multielement Analysis of Industrial Hygiene Samples," NIOSH Internal Report, presented at the American Industrial Hygiene Conference, Portland, Oregon (May 1981).
- [2] NIOSH Manual of Analytical Methods, 2nd ed., V. 7, P&CAM 351, U.S. Department of Health and Human Services, Publ. (NIOSH) 82-100 (1981).
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- [4] Ibid, V. 2, S5 (Manganese), U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).
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- [8] DataChem Laboratories, NIOSH Sequence 7998-J (NIOSH/DPSE, unpublished, April 12, 1994).
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- [10] DataChem Laboratories in-house procedure for microwave sample digestion.
- [11] Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd Ed; U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. U.S. Government Printing Office: Washington, DC, SW-846 (1988).
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- [16] Binstock, D.A., Yeager, W.M., Grohse, P.M. and Gaskill, A. Validation of a Method for Determining Elements in Solid Waste by Microwave Digestion, Research Triangle Institute Technical Report Draft, RTI Project Number 321U-3579-24, prepared for the Office of Solid Waste, U.S. Environmental Protection Agency, Washington, DC 20460 (November, 1989).

METHOD WRITTEN BY:

Mark Millson, NIOSH/DPSE, and R. DeLon Hull, Ph.D., NIOSH/DBBS.

James B. Perkins, David L. Wheeler, and Keith Nicholson, DataChem Laboratories, Salt Lake City, UT, prepared the microwave digestion procedure in the Appendix.

TABLE 1. PROPERTIES AND SAMPLING VOLUMES

Element (Symbol)	Properties		Air Volume, L @ OSHA PEL	
	Atomic Weight	MP, °C	MIN	MAX
Silver (Ag)	107.87	961	250	2000
Aluminum (Al)	26.98	660	5	100
Arsenic (As)	74.92	817	5	2000
Beryllium (Be)	9.01	1278	1250	2000
Calcium (Ca)	40.08	842	5	200
Cadmium (Cd)	112.40	321	13	2000
Cobalt (Co)	58.93	1495	25	2000
Chromium (Cr)	52.00	1890	5	1000
Copper (Cu)	63.54	1083	5	1000
Iron (Fe)	55.85	1535	5	100
Lithium (Li)	6.94	179	100	2000
Magnesium (Mg)	24.31	651	5	67
Manganese (Mn)	54.94	1244	5	200
Molybdenum (Mo)	95.94	651	5	67
Sodium (Na)	22.99	98	13	2000
Nickel (Ni)	58.71	1453	25	1000
Phosphorus (P)	30.97	44	50	2000
Lead (Pb)	207.19	328	1250	2000
Platinum (Pt)	195.09	1769	13	2000
Selenium (Se)	78.96	217	5	2000
Tellurium (Te)	127.60	450	25	2000
Titanium (Ti)	47.90	1675	5	100
Thallium (Tl)	204.37	304	25	2000
Vanadium (V)	50.94	1890	5	2000
Yttrium (Y)	88.91	1495	5	1000
Zinc (Zn)	65.37	419	5	200
Zirconium (Zr)	91.22	1852	5	200

ELEMENTS (ICP): METHOD 7300, Issue 2, dated 15 August 1994 - Page 6 of 10

TABLE 2. EXPOSURE LIMITS, CAS #, RTECS

Element (Symbol)	CAS #	RTECS	Exposure Limits, mg/m ³ (Ca = carcinogen)		
			OSHA	NIOSH	ACGIH
Silver (Ag)	7440-22-4	VW3800000	0.01 (dust, fume, metal)	0.01 (metal, soluble)	0.1 (metal) 0.01 (soluble)
Aluminum (Al)	7429-90-5	BD0330000	15 (total) 5 (respirable)	5	10 (dust) 5 (fume)
Arsenic (As)	7440-38-2	CG0625000	varies	C 0.002, Ca	0.01, Ca
Beryllium (Be)	7440-41-7	DS1780000	0.002, C 0.005	0.0005, Ca	0.002, Ca
Calcium (Ca)	-	-	varies	varies	varies
Cadmium (Cd)	7440-43-8	EU9800000	0.2, C 0.5 (dust) 0.1, C 0.5 (fume)	lowest feasible, Ca	0.01 (total), Ca 0.002 (respir.), Ca
Cobalt (Co)	7440-48-4	GF8750000	0.1	0.05	0.05 (dust, fume)
Chromium (II) (Cr)	22541-79-3	GB8280000	0.5	0.5	0.5
Chromium (III) (Cr)	18065-83-1	GB8261000	0.5	0.5	0.5
Chromium (VI) (Cr)	18540-29-8	GB8282000	C 0.1	0.001 (dust)	0.05 (soluble) 0.05 (insoluble), Ca
Copper (Cu)	7440-50-8	GL5325000	1 (dust, mists) 0.1 (fume)	1 (dust) 0.1 (fume)	1 (dust, mists) 0.2 (fume)
Iron (Fe)	1309-37-1	NO7400000	10 (dust, fume)	5 (dust, fume)	5 (fume)
Lithium (Li)	-	-	-	-	-
Magnesium (Mg)	1309-48-4	OM3850000	15 (dust) as oxide 5 (respirable)	10 (fume) as oxide	10 (fume) as oxide
Manganese (Mn)	7439-96-5	OC9275000	C 5	1; STEL 3	5 (dust) 1; STEL 3 (fume)
Molybdenum (Mo)	7439-98-7	QA4680000	5 (soluble) 15 (total insoluble) 5 (respirable insol.)	5 (soluble) 10 (insoluble)	5 (soluble) 10 (insoluble)
Nickel (Ni)	7440-02-0	QR5650000	1	0.015, Ca	0.05, Ca
Lead (Pb)	7439-92-1	OF7525000	0.05	<0.1	0.15
Platinum (Pt)	7440-06-4	TP2180000	0.002	1 (metal)	1 (metal)
Selenium (Se)	7782-49-2	VS7700000	0.2	0.2	0.2
Tellurium (Te)	13494-80-8	WY2825000	0.1	0.1	0.1
Titanium (Ti)	7440-32-6	XR1700000	as TiO ₂ , 15	lowest feasible, Ca	10
TiO ₂	13463-67-7	XR2275000	as TiO ₂ , 5 (respirable)		
Thallium (Tl)	7440-28-0	XG3425000	0.1 (skin) (soluble)	0.1 (skin) (soluble)	0.1 (skin)
Vanadium (V)	7440-62-2	YW2400000	C 0.5 (respirable) as V ₂ O ₅	C 0.05	0.05 (respir.) as V ₂ O ₅
V ₂ O ₅	1314-62-1	YW1865000	C 0.1 (fume) as V ₂ O ₅		
Yttrium (Y)	7440-65-5	ZG2980000	1	1	1
Zinc (Zn)	1314-13-2	ZH4810000	5 (ZnO fume) 15 (ZnO dust) 5 (ZnO respirable)	5; STEL 10 (ZnO fume) 5; C 15 (ZnO dust)	5; STEL 10 (ZnO fume) 10 (ZnO dust)
Zirconium (Zr)	7440-67-7	ZH7070000	5	5, STEL 10	5, STEL 10

TABLE 3. MEASUREMENT PROCEDURES AND DATA ^(a).

Element	Wavelength (nm)	Instrumental LOD (ng/mL)	Sensitivity (Intensity/ $\mu\text{g/mL}$)	Recovery		Precision (\bar{S}) (N = 3)	
				@ 2.5 $\mu\text{g}/$ filter ^(b)	@ 1000 $\mu\text{g}/$ filter	@ 2.5 $\mu\text{g}/$ filter	@ 1000 $\mu\text{g}/$ filter
Ag	328.3	28	0.65	111	91	0.02	0.075
Al	308.2	14	0.23	93	100	0.092	0.023
As	193.7	13	0.57	103	99	0.062	0.026
Be	313.0	1.5	1.29	107	90	0.040	0.034
Ca	315.9	10	0.49	99	95	0.036	0.014
Cd	226.5	1.6	0.83	107	99	0.032	0.020
Co	231.2	7.4	0.38	101	95	0.040	0.005
Cr	205.6	1.3	0.50	98	106	0.053	0.016
Cu	324.8	2.1	0.72	98	99	0.036	0.022
Fe	259.9	3.9	0.13	94	97	0.068	0.016
Li	670.8	2.8	0.48	89	95	0.171	0.043
Mg	279.6	24	0.22	105	106	0.084	0.027
Mn	257.6	0.4	0.74	84	93	0.062	0.035
Mo	281.6	7.0	0.18	94	88	0.023	0.049
Na	589.0	10	0.76	(c)	101	(c)	0.045
Ni	231.6	3.4	0.41	105	97	0.027	0.020
P	214.9	22	0.17	(c)	91	(c)	0.056
Pb	220.4	17	0.42	105	95	0.060	0.011
Pt	203.7	15	0.69	106	91	0.041	0.075
Se	190.6	21	0.28	105	97	0.068	0.049
Sn ^(a)	190.0	64	0.49	74	67	0.33	0.16
Te	214.3	29	0.41	102	94	0.050	0.063
Ti	334.9	1.2	0.55	96	106	0.051	0.029
Tl	190.9	17	0.22	103	99	0.043	0.017
V	310.2	3.2	0.88	99	94	0.043	0.014
W ^(d)	207.9	13	2.58	35	23	0.053	0.60
Y	371.0	0.8	2.35	99	100	0.015	0.013
Zn	213.9	0.6	0.60	101	94	0.013	0.013
Zr	339.2	1.9	0.88	75	98	0.049	0.008

(a) Values reported were obtained with a Jarrell-Ash Model 1160 ICP; performance may vary with instrument and should be independently verified.

(b) 2.5 $\mu\text{g}/\text{filter}$ corresponds to 5 $\mu\text{g}/\text{m}^3$ for a 500-L air sample.

(c) Blank levels too high to make accurate determinations.

(d) Qualitative only because of low recovery.

APPENDIX - MICROWAVE DIGESTION FOR LEAD IN PAINT CHIPS (AND OTHER MATRICES)

This procedure is an alternative to the procedure presented in the Sample Preparation section of this method. It provides a rapid, complete acid digestion prior to analysis by flame atomic absorption (FAA), heated graphite furnace atomic absorption (HGFAA), and inductively coupled plasma spectroscopy (ICP) [10].

Apparatus and Material [11-16]

1. Microwave apparatus requirements

- a. The microwave unit provides programmable power with a minimum of 574 W and can be programmed to within ± 10 W of the required power.
- b. The microwave unit cavity is corrosion resistant as well as ventilated. All electronics are protected against corrosion for safe operation.
- c. The system requires Teflon PFA digestion vessels (120-mL capacity) capable of withstanding pressures up to 7.5 ± 0.7 atm (110 ± 10 psi) and capable of controlled pressure relief at pressures exceeding 7.5 ± 0.7 atm (110 ± 10 psi).
- d. A rotating turntable is employed to ensure homogeneous distribution of microwave radiation within the unit. The speed of the turntable should be a minimum of 3 rpm.
- e. A safety concern relates to the use of sealed containers without pressure relief valves in the unit. Temperature is the important variable controlling the reaction. Pressure is needed to attain elevated temperatures but must be safely contained [12].
- f. Polymeric volumetric ware in plastic (Teflon or polyethylene), 50- or 100-mL capacity.
- g. Disposable polypropylene filter funnel.
- h. Analytical balance, 300-g capacity, and minimum ± 0.001 g.

Reagents

1. Nitric acid, concentrated, spectroscopy grade.
2. Reagent Water. Reagent water shall be interference free. All references to water in the method refer to reagent water that meets the ASTM Type 2 standard.

Procedure

1. Calibration of Microwave Equipment

Calibrate microwave equipment in accordance with manufacturer's instructions. If calibration instructions are not available, see EPA Method 3051 [11].

2. All digestion vessels and volumetric ware must be carefully acid washed and rinsed with reagent water. All digestion vessels should be cleaned by leaching with hot (1:1) nitric acid for a minimum of fifteen minutes, rinsed with reagent water, and dried in a clean environment.

3. Sample Digestion

- a. Tare the Teflon PFA digestion vessel.
- b. Weigh out 0.1 g paint chip sample to the nearest 0.001 g into the tared Teflon PFA sample vessel. With large paint chip samples, measure out a 2 cm² piece, weigh to the nearest 0.001 g, and quantitatively transfer it to the vessel.

- c. Add 5.0 ± 0.1 mL concentrated nitric acid to the sample vessel in a fume hood. If a vigorous reaction occurs, allow the reaction to stop before capping the vessel. Cap the vessel and torque the cap to 12 ft-lb (16 N-m) according to the manufacturer's directions. The sample vessel may be connected to an overflow vessel using Teflon PFA connecting tubes. Place the vessels in the microwave carousel. Connect the overflow vessels to the center well of the unit.
- d. Place the vessels evenly distributed in the turntable of the microwave unit using groups of two, six, or 12 sample vessels. Any vessels containing 5 mL of nitric acid for reagent blank purposes are counted as sample vessels. When fewer than the recommended number of samples are to be digested, i.e., three samples plus one blank, the remaining vessels should be filled with 5 mL of nitric acid to achieve the full complement of vessels. This provides an energy balance since the microwave power absorbed is proportional to the total mass in the cavity [14]. Irradiate each group of samples to achieve a temperature of 180°C in five minutes at a pressure of 50 psi. Continue to irradiate to achieve a temperature of 180°C at 100 psi after 25 minutes. Continue digestion for five minutes. A sample digestion program for 12 samples is presented in Table 1.

Table 1

Program Variables for Paint Chips Sample Digestion with Nitric Acid

Stage	(1)	(2)	(3)
Power	90%	90%	0%
Pressure, psi	50	100	0
Run Time, min	10:00	20:00	05:00
Time @ P, min	05:00	15:00	00:00
Temperature	180°C	180°C	0°C
Fan Speed	100%	100%	100%
Number of Vessels:	12		
Liquid Volume per Vessel:	5 mL		
Sample Weight:	0.1 g		

If the analyst wishes to digest other than two, six, or 12 samples at a time, use different values of power as long as they result in the same time and temperature conditions.

- e. At the end of the microwave program, allow the vessels to cool for a minimum of five minutes before removing them from the microwave unit. If a loss of sample is detected (e.g., material in overflow collection vessel, liquid outside liner), determine the reason for the loss (e.g., loss of vessel seal integrity, use of a digestion time longer than 30 minutes, too large a sample, or improper heating conditions). Once the source of the loss has been corrected, prepare a new sample beginning at Section 2. If insufficient material is available for reanalysis, dilute remaining digestate and note that some sample loss may have occurred.
- f. Uncap and vent each vessel in a fume hood. Add 20 mL reagent water, then reseal vessels and shake to mix thoroughly. Transfer the sample to an acid-cleaned polyethylene bottle. If the digested sample contains particulates which may clog nebulizers or interfere with injection of the sample into the instrument, allow the sample to settle or filter it.

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Settling: Allow the sample to stand until the supernatant is clear (usually, overnight is sufficient). If it does not clear, filter the sample.

Filtration: The filtering apparatus must be thoroughly precleaned and rinsed with dilute nitric acid. Filter the sample through quantitative filter paper into a second acid-cleaned container.

The digestate is now ready for analysis for elements of interest using the appropriate method.

4. **Calculations:** Report the concentrations based on the actual weight of the original sample.

POLYCHLOROBIPHENYLS

5503

mixture: $C_{12}H_{10-x}Cl_x$
[where $x = 1$ to 10]MW: ca. 288 (42% Cl; $C_{12}H_7Cl_2$);
ca. 326 (54% Cl; $C_{12}H_6Cl_3$)

CAS: Table 1

RTECS: Table 1

METHOD: 5503, Issue 2

EVALUATION: PARTIAL

Issue 1: 15 February 1984

Revision #1: 18 August 1987

Issue 2: 15 August 1994

OSHA: 1 mg/m³ (42% Cl);
0.5 mg/m³ (54% Cl)
NIOSH: 0.001 mg/m³/10 h (carcinogen)
ACGIH: 1 mg/m³ (42% Cl) (skin)
0.5 mg/m³ (54% Cl) (skin)

PROPERTIES: 42% Cl: BP 325 to 366 °C; MP -19 °C;
d 1.38 g/mL @ 25 °C;
VP 0.01 Pa (8 x 10⁻³ mm Hg;
1 mg/m³) @ 20 °C
54% Cl: BP 365 to 390 °C; MP 10 °C;
d 1.54 g/mL @ 25 °C; VP
0.0004 Pa (3 x 10⁻³ mm Hg;
0.05 mg/m³) @ 20 °C

SYNONYMS: PCB; 1,1'-biphenyl chloro; chlorodiphenyl, 42% Cl (Aroclor 1242); and 54% Cl (Aroclor 1254)

SAMPLING		MEASUREMENT	
SAMPLER: FILTER + SOLID SORBENT (13-mm glass fiber + Florisil, 100 mg/50 mg) FLOW RATE: 0.05 to 0.2 L/min or less VOL-MIN: 1 L @ 0.5 mg/m ³ -MAX: 50 L SHIPMENT: transfer filters to glass vials after sampling SAMPLE STABILITY: unknown for filters; 2 months for Florisil tubes [1] BLANKS: 2 to 10 field blanks per set		TECHNIQUE:	GAS CHROMATOGRAPHY, ECD (⁶³ Ni)
		ANALYTE:	polychlorobiphenyls
		DESORPTION:	filter + front section, 5 mL hexane; back section, 2 mL hexane
		INJECTION VOLUME:	4-μL with 1-μL backflush
		TEMPERATURE-INJECTION:	250 to 300 °C
ACCURACY RANGE STUDIED: not studied BIAS: none identified OVERALL PRECISION (δ_m): not evaluated ACCURACY: not determined		-DETECTOR:	300 to 325 °C
		-COLUMN:	180 °C
		CARRIER GAS:	N ₂ , 40 mL/min
		COLUMN:	glass, 1.8 m x 2-mm ID, 1.5% OV-17/1.95% QF-1 on 80/100 mesh Chromosorb WHP
		CALIBRATION:	standard PCB mixture in hexane
		RANGE:	0.4 to 4 μg per sample [2]
		ESTIMATED LOD:	0.03 μg per sample [2]
		PRECISION (δ_p):	0.044 [1]

APPLICABILITY: The working range is 0.01 to 10 mg/m³ for a 40-L air sample [1]. With modifications, surface wipe samples may be analyzed [3,4].

INTERFERENCES: Chlorinated pesticides, such as DDT and DDE, may interfere with quantification of PCB. Sulfur-containing compounds in petroleum products also interfere [5].

OTHER METHODS: This method revises methods S120 [6] and P&CAM 244 [1]. Methods S121 [7] and P&CAM 253 [8] for PCB have not been revised.

REAGENTS:

1. Hexane, pesticide quality.
2. Florisil, 30/48 mesh sieved from 30/60 mesh. After sieving, dry at 105 °C for 45 min. Mix the cooled Florisil with 3% (w/w) distilled water.
3. Nitrogen, purified.
4. Stock standard solution of the PCB in methanol or isooctane (commercially available).*

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: 13-mm glass fiber filter without binders in a Swinnex cassette (Cat. No. SX 0001300, Millipore Corp.) followed by a glass tube, 7 cm long, 6-mm OD, 4-mm ID containing two sections of 30/48 mesh deactivated Florisil. The front section is preceded by glass wool and contains 100 mg and the backup section contains 50 mg; urethane foam between sections and behind the backup section. (SKC 226-39, Supelco ORBO-60, or equivalent) Join the cassette and Florisil tube with PVC tubing, 3/8" L x 9/32" OD x 5/32" ID, on the outlet of the cassette and with another piece of PVC tubing, 3/4" L x 5/16" OD x 3/16" ID, complete the union.
2. Personal sampling pump, 0.05 to 0.2 L/min, with flexible connecting tubing.
3. Tweezers.
4. Vials, glass, 4- and 7-mL with aluminum or PTFE-lined caps
5. Gas chromatograph, electron capture detection (^{63}Ni), Integrator and column (page 5503-1).
6. Volumetric flasks, 10-mL and other convenient sizes for preparing standards.
7. Syringe, 10- μL .

SPECIAL PRECAUTIONS: Avoid prolonged or repeated contact of skin with PCB and prolonged or repeated breathing of the vapor [9-11].

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Break the ends of the Florisil tube immediately before sampling. Connect Florisil tube to Swinnex cassette and attach sampler to personal sampling pump with flexible tubing.
3. Sample at an accurately known flow rate between 0.05 and 0.2 L/min for a total sample size of 1 to 50 L.
NOTE: At low PCB concentrations, the sampler was found to be efficient when operated at flow rates up to 1 L/min, for 24 hours [4]. Under these conditions, the limit of detection was 0.02 $\mu\text{g}/\text{m}^3$.
4. Transfer the glass fiber filters to 7-mL vials. Cap the Florisil tubes with plastic (not rubber) caps and pack securely for shipment.

SAMPLE PREPARATION:

5. Place the glass wool and 100-mg Florisil bed in the same 7-mL vial in which the filter was stored. Add 5.0 mL hexane.
6. In a 4-mL vial, place the 50-mg Florisil bed including the two urethane plugs. Add 2.0 mL hexane.
7. Allow to stand 20 min with occasional agitation.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least six working standards over the range 10 to 500 ng/mL PCB.
 - a. Add known amounts of stock standard solution to hexane in 10-mL volumetric flasks and dilute to the mark.
 - b. Analyze together with samples and blanks (steps 11 and 12).
 - c. Prepare calibration graph (sum of areas of selected peaks vs. ng PCB per sample).
9. Determine desorption efficiency (DE) at least once for each lot of glass fiber filters and Florisil used for sampling in the calibration range (step 8). Prepare three tubes at each of five levels plus three media blanks.
 - a. Remove and discard back sorbent section of a media blank Florisil tube.
 - b. Inject known amounts of stock standard solution directly onto front sorbent section and onto a media blank filter with a microliter syringe.
 - c. Cap the tube. Allow to stand overnight.
 - d. Desorb (steps 5 through 7) and analyze together with working standards (steps 11 and 12).
 - e. Prepare a graph of DE vs. μg PCB recovered.
10. Analyze three quality control blind spikes and three analyst spikes to ensure that the calibration graph and DE graph are in control.

MEASUREMENT:

11. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 5503-1. Inject sample aliquot manually using solvent flush technique or with autosampler.

NOTE 1: Where individual identification of PCB is needed, a procedure using a capillary column may be used [12].

NOTE 2: If peak area is above the linear range of the working standards, dilute with hexane, reanalyze and apply the appropriate dilution factor in calculations.
12. Sum the areas for five or more selected peaks.

CALCULATIONS:

13. Determine the mass, μg (corrected for DE) of PCB found on the glass fiber filter (W) and in the Florisil front (W_f) and back (W_b) sorbent sections, and in the average media blank filter (B) and front (B_f) and back (B_b) sorbent sections.

NOTE: If $W_b > W_f/10$, report breakthrough and possible sample loss.
14. Calculate concentration, C , of PCB in the air volume sampled, V (L):

$$C = \frac{(W + W_f + W_b - B - B_f - B_b)}{V}, \text{ mg/m}^3.$$

EVALUATION OF METHOD:

This method uses 13-mm glass fiber filters which have not been evaluated for collecting PCB. In Method S120, however, Aroclor 1242 was completely recovered from 37-mm glass fiber filters using 15 mL Isooctane [8,13,14]. With 5 mL of hexane, Aroclor 1016 was also completely recovered from 100-mg Florisil beds after one-day storage [1]. Thus, with no adsorption effect likely on glass fiber filters for PCB, 5 mL hexane should be adequate to completely extract PCB from combined filters and front sorbent sections. Sample stability on glass fiber filters has not been investigated. Breakthrough volume was >48 L for the Florisil tube at 75% RH in an atmosphere containing 10 mg/m^3 Aroclor 1016 [1].

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METHOD REVISED BY:

James E. Arnold, NIOSH/DPSE; S120 originally validated under NIOSH Contract 210-76-0123.

Table 1. General Information.

<u>Compound</u>	<u>CAS</u>	<u>RTECS</u>
Polychlorinated Biphenyls	1336-36-3	TQ1350000
Chlorobiphenyl	27323-18-8	DV2063000
Aroclor 1016 (41% Cl)	12674-11-2	TQ1351000
Aroclor 1242 (42% Cl)	53469-21-9	TQ1356000
Aroclor 1254 (54% Cl)	11097-69-1	TQ1360000

Table 2. Composition of some Aroclors [15].

<u>Major Components</u>	<u>Aroclor 1018</u>	<u>Aroclor 1242</u>	<u>Aroclor 1254</u>
Biphenyl	0.1%	<0.1%	<0.1%
Monochlorobiphenyls	1	1	<0.1
Dichlorobiphenyls	20	16	0.5
Trichlorobiphenyls	57	49	1
Tetrachlorobiphenyls	21	25	21
Pentachlorobiphenyls	1	8	48
Hexachlorobiphenyls	<0.1	1	23
Heptachlorobiphenyls	none detected	<0.1	6
Octachlorobiphenyls	none detected	none detected	none detected